

HIGH TEMPERATURE THERMOCOUPLE
RESEARCH AND DEVELOPMENT PROGRAM

MONTHLY PROGRESS REPORT NUMBER 12
Period 1 May 1964 to 1 June 1964
Contract Number NAS 8-5438
Request Number TP 3-83547

prepared for
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Huntsville, Alabama

work performed by
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ABSTRACT

This report covers the period 1 May 1964 to 1 June 1964, under Contract NAS 8-5438, which calls for twelve months of research and development of a high temperature thermocouple capable of measuring rocket engine exhaust temperatures in the 3000°C range, under adverse conditions of oxidation, erosion, vibration and shock. This is the last monthly progress report to be submitted.

The primary objectives of the program are to advance the state-of-the-art of high temperature thermometry and to develop an end product suitable for in-flight temperature measurements on the SATURN vehicle.

Vibration tests to 60g on the sheath materials were performed without failure. The search for better oxidation resistant coatings was continued without success. Preparation of the final engineering report for this program was started. The final three Type 4735 gages were prepared for assembly, but final assembly was halted pending a review of tests conducted by Southern Research Institute.

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SECTION ISUMMARY1.0 Period Covered

This report covers the period 1 May 1964 to 1 June 1964.

1.1 Statement of Work

The Contractor shall advance the state-of-the-art of high temperature thermometry and specifically improve the technique of accurately measuring high temperatures by designing, fabricating, testing, and delivering nine (9) thermocouple probes capable of operation in the 3000°C temperature range under adverse conditions of erosion oxidations and high stress levels for useful period of time. Also, present methods of thermocouple probe fabrication will be modified such that the end product will be suitable for in-flight temperature measurements on the SATURN vehicle.

To accomplish the above objectives the Contractor shall consider and explore specific R & D efforts as follows:

- a. Develop of the physical structure of an immersed probe to attain minimum drag and highest resistance to bending and shear forces.
- b. Ascertain the best combination of ingredients in the protective coating of the probe to extend the term of oxidation resistance.
- c. Determine the best combination of compensated lead wires for use with the immersion type probes.
- d. Incorporate latest state-of-the-art materials as potting and sealing elements in the base of the probe.
- e. Determine effects of reactions between oxide coatings and tungsten in relation to the emf output.

1.1 Statement of Work Cont....

- f. Establishment of rates of erosion for different types of refractory coatings such as tungsten disilicide, carbides and cermets when subjected to high velocity, high temperature gas streams.

1.2 Progress

a. Protective Coatings

No oxidation resistant coating better than those being used has been found.

b. Vibration Tests

Two sheaths were vibration tested to 60g without adverse effects.

c. Final Report

Work was started on preparation of the final report.

d. Calibrations, Low Temperature

ACL low temperature calibrations were verified by Hoskins Manufacturing Company.

e. Calibrations, High Temperature

Information was transmitted to ACL that SRI has reported adversely on ACL high temperature calibrations. Plans were made for a thorough investigation of the SRI data.

SECTION II

PAST PROGRESS

2.0 General

Previous effort was reported in ACL Progress Reports T-1097-1 through T-1097-11.

2.1 Prototype Design and Development

As was previously reported, objectives for the first prototypes were limited to the 4000°F - 4500°F range in the interest of accumulating test data for analysis, the results to be utilized in future design.

A design approach for the prototype gauges was selected, and drawings prepared, detailing means of fabrication and assembly.

Investigations made into fabrication techniques involved in working vapor deposited Tungsten, resulted in improved material handling techniques.

Shock and vibration tests, performed on a prototype mock-up, resulted in a conclusion that the sheath material was intrinsically capable of withstanding the specified shock and vibration requirements.

Samples of various types of compensation lead wires were ordered for test and evaluation.

An evaluation of the SRI calibration tests for ACL Type 4734 gauges was made, resulting in a conclusion that an optimum immersion depth might be in the order of 1 - 1/2 inches in an isothermal region.

The two Type 4734 gauges tested by N.A.S.A., and returned to ACL were examined and results of the examination were reported.

2.1 Prototype Design and Development Cont....

A test of a "no-insulation" approach was started, but was aborted due to a failure in the test oven. Such tests were subsequently continued.

Three prototype gauges were delivered to M-ASTR-I, on 17 October 1963, for test and evaluation. Calibration of this type of gauge indicated a shift in emf output to a higher value than that shown in previous calibrations. The shift was believed due to a spurious emf contributed by the "compensated" lead wires. The curves, however, paralleled the curves taken by Southern Research Institute, as well as those predicted by ACL.

Further tests verified the presence of lead wire errors.

Analyses of form and shock drag loads were made. The results will be considered in future design.

Investigations of oxidation resistant coatings were continued. Accumulated data was reviewed, and tabulated for comparison and reference.

Response test performed on one Type 4735 gauge yielded response as low as 45 milliseconds from ambient air to boiling water. Lead wire tests resulted in a conclusion that the thermocouple materials should be used in lead extensions for best accuracy. Further investigations of oxidation resistant coatings, and insulators verified the conclusion reached in earlier tests. Design of the second generation gauges was continued.

Three second generation gauges were delivered to M-ASTR-I on 26 February 1964 for test and evaluation. These gauges incorporated thermocouple materials as lead wire, and elimination of BEO insulation in the Hot Zone. Calibrations of this type of gauge showed an increase in the upper temperature limit, and virtual elimination of lead wire error. emf output curves essentially tracked predicted values.

Continuation of calibration tests resulted in verification of calibration curves previously developed.

2.1 Prototype Design and Development Cont....

Further calibration and stability tests were highly repeatable. A positive drift, diminishing in extent with cycling, was noted.

Calibrations in the over 4000°F range were continued and were in excellent agreement with those previously made. Low temperature calibrations to -320°F were performed. Vibration tests on the sheath materials were performed without failures. Materials tests on surface-treated sheath materials resulted in an increase of 50% in strength. Work was started on the final engineering report for this project.

SECTION III

CURRENT PROGRESS

3.0 General

ACL has directed effort in the current reporting period toward continuance of vibration testing, work on the final engineering report, and preparation for delivery of the final three gauges required under this contract.

At the end of the reporting period, ACL was advised of adverse results of SRI evaluation tests which are in wide disagreement with results of ACL tests as well as tests performed by others. This is discussed in more detail in this section of the report.

3.1 Progress

3.1.1 Vibration Tests

Vibration testing had been performed on the Type 4735 gauges to limited (14g) input levels, as discussed in previous reports. In order to more fully establish the characteristics of the sheath materials of the Type 4735 gauges within scope of work requirements, two additional tests were performed to sinusoidal input levels of 60 G, without failures, which exceeds the objective of 50 G by 10 G. The test articles, methods, and results are described below.

3.1.1.1 Test Articles

Two test sheaths, each of different construction, were fabricated of vapor deposited tungsten, using the same techniques of construction as in the Type 4735 gauges. They are described separately as follows, as Gauge "A" and "B" for identification.

3.1.1.2 Gauge "A"

Gauge "A" was made with a biconvex cross section, 0.103 inch in the minor axis, and 0.258 inch in the major axis. Unsupported immersion length was 1.50 inches. (See Figure 1) The last inch of length from the tip was tapered $8\text{-}1/2^\circ$, in the major dimension and 1° in the minor dimension. Nominal wall thickness was .020 inch, although the wall thickness in the minor axis may have been as thin as 0.008 inch in places, because of variations in straightness prior to final dimensioning. The center conductor was W26Re, 0.020 inch in diameter, vapor deposited in the tip to form a junction with the tungsten sheath. It was unsupported over its length. This gauge had not been heated, therefore it was in the raw, or non-recrystallized condition.

3.1.1.3 Gauge "B"

Gauge "B" was made with a tubular, symmetrical cross section, 0.123 inch outside diameter, and a nominal 0.020 wall thickness. Unsupported immersion length was 1.95 inches. (See Figure 1). The last inch of length from the tip was tapered 1° . As in the Type "A" gauge, the 0.020 inch W26Re center conductor was vapor deposited in at the tip, and was unsupported over its length. This gauge had been temperature cycled from 2000°F to 4300°F at least six times, and was therefore fully recrystallized.

3.1.1.4 Test Method

The test gauges were cast in Devcon "B", a dense cement, with their unsupported immersion length extending from the cement block. The block was tightly clamped in a vibration fixture mounted on the head of a Ling electrodynamic vibrator. Calibrated accelerometers mounted on the head and the gauge base were used to measure input and output accelerations. Inputs were increased in steps of 5g to a maximum input of 60g, at the resonant points found during a logarithmic sweep resonance search.

3.1.1.5 Test Results Gauge "A"

During the scan on Gauge "A", resonances were observed as follows:

<u>Axis</u>	<u>cps Resonant Freq.</u>	<u>G Input</u>	<u>Max. G Output</u>
Minor (Y)	1496	5	150
Major (X)	1497	5 - 10	235
	1500	5 - 60	saturated
Major (X)	3268	5	260

Time at each G-input level at resonance was a minimum of 15 minutes.

No adverse effects were noted during or after this test.

3.1.1.6 Test Results, Gauge "B"

During the scan on Gauge "B", one resonance at 550 cps was noted. The gauge was subjected to inputs of 5g to 60g at resonance in 5g increments, and was vibrated at the 60 G level for 15 minutes. No adverse effects were noted during or after this test.

3.1.1.7 Discussion

As can be seen from the descriptions of the two test gauges given above, there is a considerable difference in physical characteristics between the Type 4735 gauges previously vibration tested, and Gauges "A" and "B". The fineness ratios of Gauges "A" and "B", however, are considerably larger than the Type 4735 gauge. Therefore, these tests represent worst case conditions and may be taken as a measure of the reaction of the Type 4735 gauge sheath to vibratory inputs greater than those stated in the scope of work.

The major consideration for the tests described herein, was the resolution of whether the sheath material, in the configuration used in ACL Series 4700 gauges, was suitable for use under a severe vibration environment. The question was further compounded

3.1.1.7 Discussion Cont....

by the fact that; 1) ordinary tungsten has a well established recrystallization temperature characteristic which is associated with a serious decrease in ductility, and 2) in the cold (ambient temperature) state is notoriously brittle. The vibration tests at ambient temperature therefore were performed under adverse conditions. A great deal of concern has been expressed by N.A.S.A., as well as others, whether the sheath material would fail under vibration, and it was considered necessary by ACL to prove or disprove this contention.

As a consequence of these vibration tests, it is concluded that the sheath material, vapor deposited tungsten, as fabricated by San Fernando Laboratories, Pacoima, California, to ACL design configurations, and processed by ACL, is suitable for applications such as the Type 4735 gauges.

ACL had considered, moreover, that alternate sheath types, such as the Type "A" and "B" sheaths, should be investigated in the interest of the scope of work regarding sheath configurations for minimum drag and highest resistance to bending and shear forces.

3.1.2 Calibrations, High Temperature

It had been considered by ACL that calibration of the Type 4735 gauges had been established in the range 2000°F to over 5000°F as a result of the accumulation of a large number of measurements within the range. These measurements had been made without a single failure, except for those deliberately induced during oxidation tests.

At the time of report preparation, however, during a telephone conference with M-ASTR-I personnel, it was related that two of the second generation Type 4735 gauges, being tested by Southern Research Institute, failed to perform beyond 3500°F. It is understood by ACL that the following occurred; 1) The output of the gauges appeared to deteriorate after 3500°F 2) that some material, presently unknown, appeared to melt and drip from the sheath. 3) SRI, at this point, terminated the tests.

3.1.2 Calibrations, High Temperature Cont....

The SRI report is in wide variance with results observed during ACL tests by, not only ACL technicians and observers, but by representatives of other companies as well. In fact, other ACL gauges, fabricated in a similar manner, and tested by the user, repeated the ACL curves with very close agreement, from 1500°F to about 4500°F. These independent tests were conducted by Aerojet on probes intended for use in the control loop for the NERVA propulsion system. Their calibrations were conducted under such stringent procedures, and under such extremely close observation and critical supervision by SNPO that the results were accepted without question by ACL.

The feedback information from SRI is, in ACL's opinion, so meager at this time, that no conclusions can be drawn as to the failure of the Type 4735 gauges to repeat their previously demonstrated performance. Slight variations, which could be attributed to differences in technique, experimental error, or observer error could be accepted. A major variance, however, such as that described herein, should, in ACL's opinion, be thoroughly investigated. ACL will therefore visit M-ASTR-I on 11 June 1964 to attempt to determine the facts relevant to the SRI report.

It should be pointed out, in fairness to all concerned, that this is the first knowledge that ACL has received of any inadvertence in performance of either the first generation gauges, delivered on 17 October 1963, or the second generation gauges, delivered on 26 February 1964, whereas it is stipulated in the contract that return information was to be supplied ACL within ten days of any delivery. ACL is thus in the position of having received adverse information within 15 days of the termination date of the contract, and, therefore, may not have reasonably sufficient time to incorporate any indicated design changes in the final three gauges, which were scheduled for delivery on 17 June 1964 in accordance with contract requirements.

3.1.3 Calibrations, Low Temperature

In the last report, an account was given of low temperature calibrations of the W-W26Re materials from 32°F to -320°F. A mistake in transcribing data stated a negative output of .03 millivolts at -320°F. This should have been a positive output of .03 millivolts. At ACL's request, Hoskins Manufacturing Company performed calibrations over the same temperature range

3.1.3 Calibrations, Low Temperature Cont....

and reported their findings to ACL. The graph of Figure 2 is a plot, showing both ACL and Hoskins data. Considering experimental error, it is felt there is good agreement between the two curves.

3.1.4 Final Delivery

ACL had started assembly of the three third generation Type 4735 gauges when word was received on 2 June 1964 of the results of the SRI tests. ACL accepts the possibility that the apparent malfunctions could be due to a faulty fabrication technique, although this is considered remote in light of the extensive testing performed on both identical and similar gauges. Therefore, assembly was halted until such time as the facts are ascertained. It is believed possible that investigations can be made and a conclusion drawn as to the cause of the malfunction in a timely manner. It is recommended, however, that an extension of one month be granted, in view of the circumstances. ACL will, in any event, expend every reasonable effort to deliver the best possible product in the least time.

SECTION IV

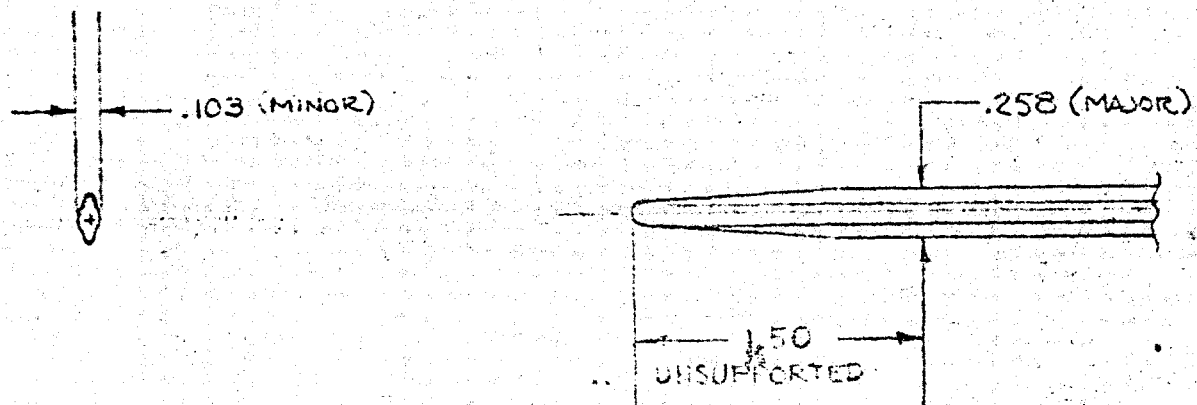
PROGRAM FOR NEXT INTERVAL

4.0 Objectives for the Interval 1 June 1964 to 17 June 1964 are:

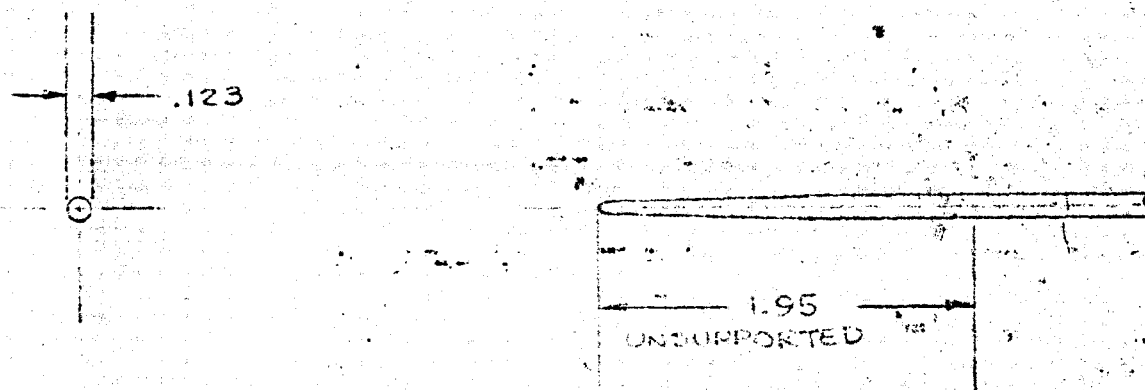
- a. Review results of SRI tests.
- b. Deliver the final three gauges on 17 June 1964, if possible.
- c. Continue work on Final Engineering Report.

SECTION VSTATEMENT OF MAN HOURS5.0 Hours by Category

<u>Category</u>	<u>Previous Periods</u>	<u>Current Period</u>	<u>To Date</u>
Engineering	831.5	121.0	952.50
Clerical	165.5	16.0	181.5
Fabrication	770.5	7.5	778.0
Consulting	20.5	- 0 -	20.5
Drafting	61.0	- 0 -	61.0



TEST GAGE "A"



TEST GAGE "B"

FIG. 1 - VIBRATION TEST GAGES

EUGENE DIETZGEN CO.
MADE IN U. S. A.

NO. 340R-10 DIETZGEN GRAPH PAPER
10 X 10 PER INCH

TEMP.
°F
+200
150
100
50
+
0
-
50
100
150
200
250
300
-350

THOMAS DATA
AGE DATE

WE WOULD ESTIMATE
FEET TO EXIST

NOTE: COLLECTED 1/14/50

5201 W. 100th ST. MINN.

LOS ANGELES 45, CA

KEITH
A. F. 1954

FIG. 2 - LOW TEMPERATURE
CALIBRATIONS

OVERALL M.V.